

LIS007879221B2

(12) United States Patent

Pütter et al.

(10) Patent No.: US 7,879,221 B2 (45) Date of Patent: Feb. 1, 2011

(54) PROCESS FOR PREPARING POROUS METAL ORGANIC FRAMEWORKS

(75) Inventors: **Hermann Pütter**, Neustadt (DE); **Markus Schubert**, Ludwigshafen (DE);

Ingo Richter, Schwetzingen (DE); Ulrich Müller, Neustadt (DE); Natalia Trukhan, Ludwigshafen (DE)

(73) Assignee: BASF SE, Ludwigshafen (DE)

(*) Notice: Subject to any disclaimer, the term of this

patent is extended or adjusted under 35 U.S.C. 154(b) by 219 days.

(21) Appl. No.: 12/300,902

(22) PCT Filed: May 11, 2007

(86) PCT No.: PCT/EP2007/054554

§ 371 (c)(1),

(2), (4) Date: **Dec. 23, 2008**

(87) PCT Pub. No.: WO2007/131948

PCT Pub. Date: Nov. 22, 2007

(65) Prior Publication Data

US 2009/0171107 A1 Jul. 2, 2009

(30) Foreign Application Priority Data

May 16, 2006 (EP) 06114002

(51) **Int. Cl.**

 C25B 3/12
 (2006.01)

 C07F 15/00
 (2006.01)

 C07F 3/00
 (2006.01)

53

536/49, 53, 61, 114, 123, 136, 147, 183; 549/3, 211; 546/225; 534/16; 205/457

See application file for complete search history.

(56) References Cited

U.S. PATENT DOCUMENTS

3,964,983 A 6/1976 Eisenbach et al.

5,648,508	A	7/1997	Yaghi	
2007/0227898	A1*	10/2007	Muller et al	205/457
2008/0214806	A1	9/2008	Schubert et al.	
2009/0183996	A1*	7/2009	Richter et al	205/424

FOREIGN PATENT DOCUMENTS

DE	10 2005 023 856	5/2005
WO	WO-02088148	11/2002
WO	WO-2005/049892	6/2005
WO	WO-2006/125761	11/2006

OTHER PUBLICATIONS

Eddaoudi et al., "Systematic Design of Pore Size and Functionality in Isoreticular MOFs and Their Application in Methane Storage," Science, vol. 295 (Jan. 18, 2002, pp. 469-472).

Chen et al., "Interwoven Metal-Organic Framework on a Periodic Minimal Surface with Extra-Large Pores," Science vol. 291 (Feb. 9, 2001), pp. 1021-1023.

Seki et al., "Syntheses and Characterization of Microporous Coordination Polymers with Open Frameworks," J. Phys. Chem. B 2002, 106, pp. 1380-1385.

S. Kitagawa et al., "Functional Porous Coordination Polymers," Angew. Chem. Int. Ed. 43 (2004), pp. 2334-2375.

International Preliminary Report on Patentability issued Jan. 13, 2009 in corresponding International Application No. PCT/EP2007/054554, along with the English language translation.

* cited by examiner

Primary Examiner—Porfirio Nazario Gonzalez (74) Attorney, Agent, or Firm—Connolly Bove Lodge & Hutz LLP

(57) ABSTRACT

The present invention relates to a process for preparing a porous metal organic framework comprising at least two organic compounds coordinated to at least one metal ion, which comprises the steps

- (a) oxidation of at least one anode comprising the metal corresponding to at least one metal ion in a reaction medium in the presence of at least one first organic compound which is an optionally substituted monocyclic, bicyclic or polycyclic saturated or unsaturated hydrocarbon in which at least two ring carbons have been replaced by heteroatoms selected from the group consisting of N, O and S to form a reaction intermediate comprising the at least one metal ion and the first organic compound; and
- (b) reaction of the reaction intermediate at a prescribed temperature with at least one second organic compound which coordinates to the at least one metal ion, with the second organic compound being derived from a dicarboxylic, tricarboxylic or tetracarboxylic acid.

10 Claims, No Drawings